PRELIMINARY INVESTIGATION FOR A CONTINUOUS HYDROGENATION OF VEGETABLE OIL

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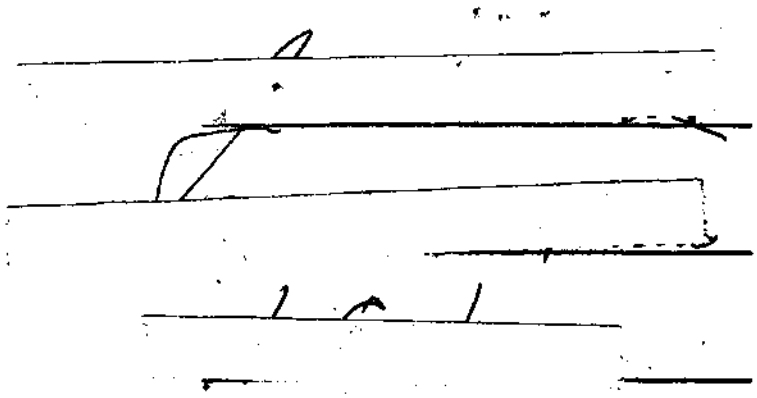

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DEDICATION

This work is sincerely dedicated to my wife, Ellen, without whose constant aid in the home and in the laboratory this thesis would have never reached its present form.

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INTRODUCTION

The need for a satisfactory continuous process for the hydrogenation of vegetable oils has been apparent for many years and is becoming more and more important with the increased consumption of these oils. Although efficient continuous processes have been available for some time for the other unit processes involved in the manufacture of edible oil products, the hydrogenation phase remains unsolved.

This thesis is intended to be a preliminary investigation of a possible continuous hydrogenation process. Because of the nature of the process, it was necessary to design apparatus on a somewhat larger scale than the usual bench model. In view of the size of the apparatus and the fact that this particular investigation was entirely new, it was anticipated that many unforeseen problems would arise. Most of these problems have been solved but others await further study.

HISTORICAL DEVELOPMENT

Because of the extensive amount of material published on hydrogenation, a complete review of the literature in this work is impractical. For the past fifty years scarcely a day has passed without some additional paper or publication on this subject -- certainly the total publications must number into the thousands.

A brief summary of the history of catalytic hydrogenation and a somewhat more comprehensive discussion of efforts toward continuous processes, with emphasis on the British TRW process, will be given.

It may be said that the recorded history of catalytic chemistry began in 1794 when a Mrs. Fulhame published "An Essay on Combustion" which dealt with the influence of water on chemical reactions.[1] The nineteenth century therefore became the incubation period of catalysis with the actual birth taking place just at the turn of the century.

It was not until the year 1897 that any systematic study of the catalytic properties of metals was made. Sabatier and Senderens in 1900 published the results of their researches in this direction. They showed through their experiments that absorption of hydrogen occurred when the vapor of fatty acids together with hydrogen were brought into contact with a suitable metallic catalyst. They found after many experiments that nickel most nearly approached the ideal metal to be used. The treating of the vapors of fatty substances was found to be of no practical value because the glycerides decomposed when subjected to the high temperatures required for vaporization.

It was not until 1902 that any proposal was made to hydrogenate the oil in its liquid form, this being made by Leprince and Siveke.[2] In 1903 a patent was issued in England to Normann[3] which placed the business of vegetable and animal oil hydrogenation on a firm commercial footing.

Shortly after the announcement of Normann's application to the liquid phase hydrogenation of oils a number of researchers and inventors put their talents to work toward the improvement of industrial technique. Many designs of industrial apparatus were proposed and a few realized actual application. It seemed, however, that the general trend of these "improvements" was to more and more complicated design with little or no actual improvement in the hydrogenation characteristics.

A British patent to Bedford and Williams in 1907[4] described a method of exposing oil to the action of hydrogen by treating the oil in films in an atmosphere of hydrogen and in contact with a nickel catalyst.

Subsequent inventors described methods of injecting the oil and catalyst slurry as a spray into a hydrogen atmosphere, spraying the oil through a hydrogen atmosphere onto a fixed catalyst bed, spraying with countercurrent oil-hydrogen streams and other innovations. Others patented detailed mechanical mixers to effect good contact between the oil, catalyst, and hydrogen. Still others delved into complicated baffle systems, atomizers, perforated buckets, spiral propellers and other similar devices.

With the passage of time, however, practically all of these schemes have been discarded. By far the majority of hardening plants

today utilize the agitation medium implied by Normann in his original patent of 1903: simply bubbling hydrogen through a tank of preheated oil-catalyst mixture. Contemporary vessels may or may not be equipped with mechanical paddle-type agitators.

One of the first attempts at continuous hydrogenation was described by Moore in 1914.[5] His apparatus was a double-compartment type vessel with a filtering diaphram containing the catalyst between the two sides. Oil is injected through a nozzle into the top compartment where it meets a blast of hydrogen and is atomized. The oil and hydrogen mixture then penetrates the catalyst bed and passes to the lower chamber.

A series of spray chambers originally patented by Testrup in 1901[6] proposes a method for continuous operation but it was never used commercially. A rather complicated arrangement of concentric tubes and slide dampers described by MacDougall in 1921[7] was said to be applicable to continuous operation but never passed beyond the pilot plant stage.

The first successful attempt at continuous hydrogenation was developed by Bolton and Lush for The Technical Research Works, Ltd.[8] Hydrogenation is carried out in a number of drawn steel-jacketed tubes, arranged in series. Each tube is approximately seven feet long and six inches in internal diameter. The cages are built of Monel metal gauze and are filled with nickel turnings whose surface has been activated. Oil to be hardened is contained in a tank, fitted with heating coils, sight and pressure gages, etc., which is connected to one end of the series of tubes. At the other end is a separator

and a continuous cooling coil. The working pressure is 150 pounds gage.

The catalyst cages are activated electrolytically by covering the metal with a thin film of nickel oxide. The cages are immersed in dilute sodium carbonate solution and surrounded by a thin sheet of nickel, which forms the cathode, the cage itself being the anode. From 24 to 48 hours are required for the necessary oxidation. The cages are then washed and placed in the reaction tubes. The tubes are closed, superheated steam is turned into the jackets, and the oxide is reduced to active nickel by a stream of hydrogen.

Oil to be hardened is pumped into the feed tank, is preheated, and when the desired temperature is attained, it is forced by hydrogen pressure from the feed tank into the main supply line where it meets the current of hydrogen. Oil and hydrogen pass together into the top of the first tube, and the oil drips through the catalyst to the bottom, is forced to the top of the second tube, and soon through the whole series, being at all times in contact with hydrogen.

At the end of the line, the oil is received in the separator, when it is freed from excess hydrogen, and is then cooled continuously in the cooling coil to a suitable temperature for discharge to the atmosphere. Filtration of the oil is not necessary since the catalyst is not suspended in it.

The largest plant offered is rated at a capacity of only 33,000 pounds of hardened cottonseed oil with a melting point of 40°-42°C per day.[9] This, together with the fact that the massive

catalyst employed is not so selective as those employed in batch operation, has retarded the adoption of this process in the United States.

NATURE OF THE REACTION

Normal hydrogenation of vegetable oils consists simply of adding a molecule of hydrogen to an ethylenic bond of a fatty acid chain. Despite the enormous amount of research which has been done, the exact mechanism of the reaction is not known. Several theories have been proposed to explain the various steps in the overall reaction, but none of them have been proven to complete satisfaction. It is generally assumed that some sort of adsorption complex is formed with the oil at the ethylenic bond on the surface of the catalyst. This complex then reacts with the hydrogen, saturating the ethylenic linkage and reactivating the catalyst surface with accompanying liberation of heat. Triply unsaturated bonds are limited to marine oils and the drying oils, so are not of concern here.

When saturated glycerides are the desired end product, no complications arise as the resultant reaction gives merely the saturated side chains. For vegetable oils this end result would be principally stearic acid chains as the C_{18} acids are almost universally prevalent. However, as soon as fatty acid chains with more than one ethylenic bond are considered, the difficulties become more involved. Cottonseed oil contains approximately 23 per cent oleic acid having one double bond and 48 per cent linoleic[10] with two double bonds with small percentages of other monoethanoid acids, and as hydrogenation for edible purposes implies only partial saturation of the molecule, several questions arise: Are the bonds saturated in any definite order? Are any side reactions induced? What isomers, if any, are encountered?

On the basis of the experimental evidence of many researchers, it appears that preferential saturation does occur. This preferential addition is termed "selectivity" and is a very important factor in the final quality of processed oil for edible purposes. In order to suppress rancidity it is the goal of hydrogenation to reduce the iodine value as much as possible and still maintain a satisfactory melting point of the product. Linoleic acid is unsaturated at the 9-10 and 12-13 positions. If the 9-10 linkage is saturated the resulting 12-13 iso-oleic acid will have a melting point of about 40°C, but if the 12-13 position is saturated normal oleic acid with a melting point of about 15°C will be formed.[11] It is obvious that normal oleic is the result to be desired.

The question of selectivity is largely a matter of the catalyst used. Research on production of highly selective catalysts has led to the reduction of iodine values of all vegetable shortening from about 90 in 1920[12] to about 68 in 1948. It has been found, however, that temperature also affects selectivity, lower reaction temperatures generally producing greater selectivity. Because of the decreased reaction velocity at the lower temperatures, it is the usual industrial practice to sacrifice some of the selective properties of the reaction in favor of the decreased reaction time. Temperature ranges of about 300°-360°F are of common practice.

Selectivity refers not only to the preferential hydrogenation of one of two possible ethylenic bonds within the same fatty acid chain but also to the conversion of linoleic chains to oleic before

the oleic is saturated to Stearic. Bailey[13] has indicated the path of hydrogenation ideally and with a good selective catalyst. Thus, the ultimate goal of the catalyst and other variables is to eliminate the linoleic acid at an iodine value of 66. Such a product would have maximum stability and "keeping" properties.

The overall reaction is much more complicated than has been presented thus far. There have been strong evidences that oleic acids are produced with the double bond at points other than those of the linoleic acid. This situation would indicate a continual hydrogenation-dehydrogenation type of reaction occurring throughout the process.

In addition to the positional isomers involved, the reaction is further complicated by geometric isomers, for wherever a double bond occurs a cis-trans isomer is possible, and usually encountered. For a thorough treatment of isomers involved in the reaction the reader is referred to the works of Bailey,[14] Markley,[15] and Ellis.[16]

EQUIPMENT AND ACCESSORIES

Process piping used consisted of 47 feet of schedule 40 1-1/2 inch steel pipe. Two 21-foot lengths were arranged horizontally, one above the other, on a pipe rack and connected at one end by a five foot vertical section. The oil was discharged from the end of the upper length into a cylindrical surge tank 16 inches in diameter and 10 inches high. A 3/4 inch purge line was installed in the top of the surge tank to vent to the atmosphere that hydrogen which had not reacted with the oil. From the bottom of the surge tank a 1-1/2 inch pipe led to the intake side of the pump. See Figures 1 and 2.

The pump used was a Model 20-DVM Tri-Rotor with a built-in vernier by-pass manufactured by Yale & Towne Company. This pump was chosen for its several unique characteristics which made it particularly suitable for this work. First, it is basically a positive displacement rated at 100 lb/in^2 pressure at discharge against zero head and a maximum rate of 20 gallons per minute. Secondly, the built-in by-pass fitted with a vernier scale allows the pump to be used for metering purposes, once the by-pass has been calibrated, without changing the drive speed. Thirdly, the design allows the pump to be used in continuous operation with temperatures up to 400°F. Prime mover for the pump was a 1140 RPM 1 H.P. 210 V electric motor with direct coupling to the pump drive shaft.

On the discharge side of the pump, a one foot vertical section of 1-1/2 inch pipe was installed to cushion any small pulsations in the oil flow. This was followed by a 1-1/2 inch standard 150 lb.

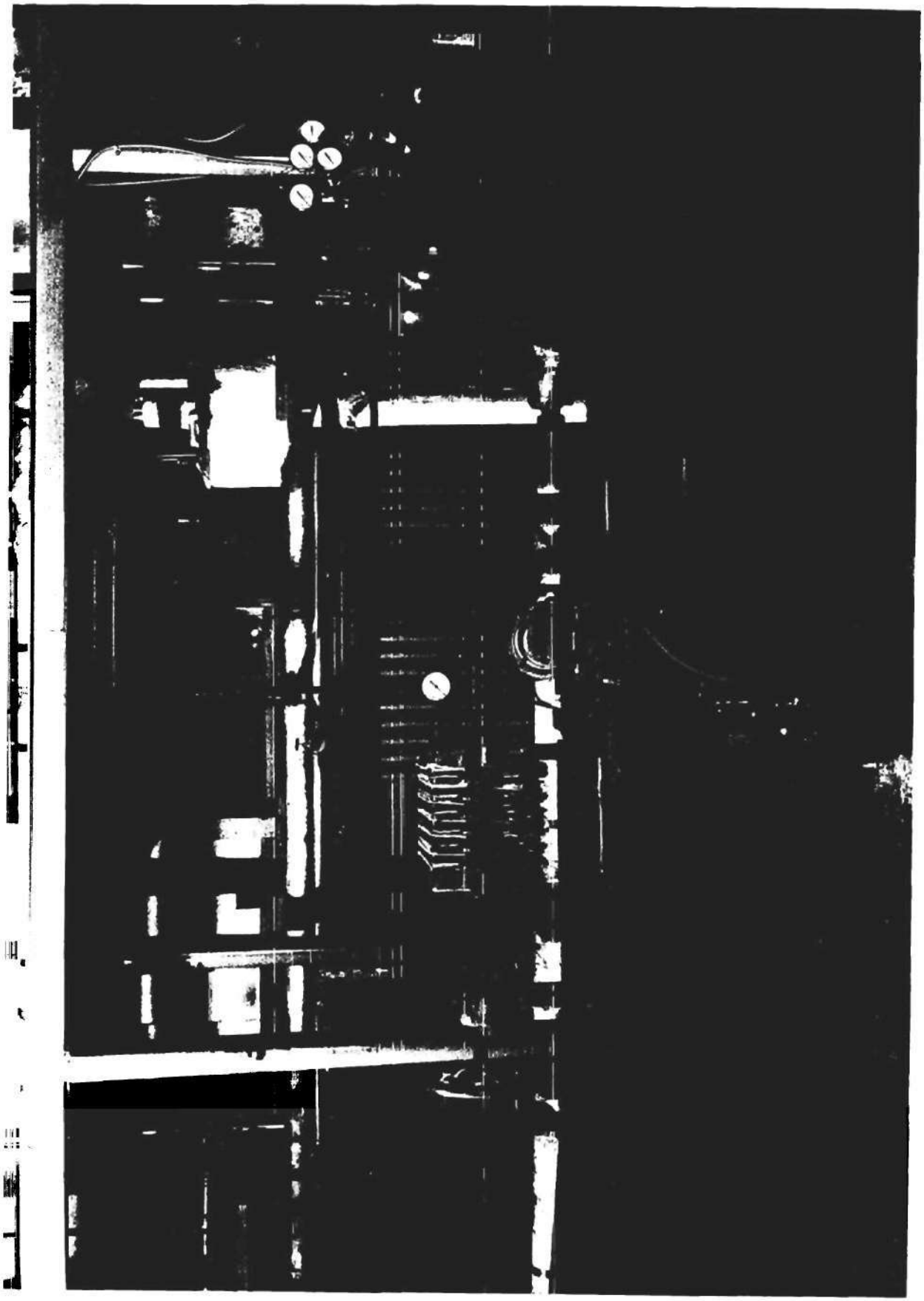

Figure 1.

Figure 2.

visible flow check valve manufactured by Schutte-Koeting. This valve served the double purpose of protecting the pump against any sudden burst of pressure from the system as well as affording a visible check on the fluid flow in the system.

Immediately beyond the check valve, the hydrogen nozzle projected through a tee and into the oil stream. The nozzle was machined from brass rod stock 3/8 inch in diameter and had a throat .015 inch in diameter. The nozzle was made in two sections to afford facility in changing outlet diameters. The external section was machined from 3/4 inch brass rod stock with standard 3/4 inch male threads on both ends, a 1/4 inch smooth hole through the entire length of six inches and one end tapped with 3/8 inch standard pipe thread. The internal section could then be screwed into the inner threads and the assembly installed into the 1-1/2 inch tee through a bushing. This arrangement made possible the replacement of different heads with the least effort so that differently designed nozzles could be tested.

Hydrogen and nitrogen were led to the nozzle through 1/4 inch steel tubing from the bottled gases. This line was fitted with a needle valve and gravity-type check valve to protect the reducing valves on the bottles from the possibility of oil backing up in the line.

A 1/2 inch side line was installed just after the nozzle and led to a six plate 10 x 10 inch plate and frame Sperry cast iron filter press. As the main pump was to be used to develop filtration pressure a cock valve was placed after the filter line. A 1-1/2 inch by-pass line was installed, with cock valves on both ends, between the

downstream side of the filter line valve and the suction side of the pump in order to allow the entire system to be drained and filtered. A recirculating line was installed from the filtrate tray of the press through a gear pump driven by a 1/2 H.P. 210 V electric motor and into the top of the surge tank.

A pressure regulating gate valve was installed at the downstream end of the process piping to regulate the system pressure for a chosen vernier setting on the pump by-pass. Steam and air inlets, for blowing purposes, were brought into the system just upstream of the pressure regulating valve through a tee and bushing.

Heat for preheating the charge was obtained with three electrical resistance heaters. The main heater, a Westinghouse Corox 2000 watt 110 V oil immersion heater, was located in the surge tank. Two auxiliary heaters, both of 650 watt output consisted of 18 gage nichrome wire wound around each of the vertical pipe sections and operated from 110 V A.C. current.

The entire system was lagged with standard one inch 85 per cent magnesia pipe covering.

Two 0-100 p.s.i.g. pressure gages were installed in the process piping, the first immediately upstream of the visible flow check valve and the second about eight inches upstream of the pressure regulating valve. Two 400°F mercury bulb thermometers were provided, through a tee at the top of the vertical section in the process piping and in the surge tank. Breakage difficulties necessitated removing the thermometer in the surge tank. A pressure gage was also installed on the inlet line to the filter press.

Both paper and canvas were used for filtration with Dicalite as the filteraid.

EXPERIMENTAL PROCEDURE AND RESULTS

Experimental Procedure

Construction on the pipe rack, made of four inch steel tube column and four inch I-beam stringers and spacers, was begun in July 1950. The structure was securely bolted to a concrete floor and all joints arc welded. Installation of the main pieces of equipment was completed in November 1950. Due to delays in receiving proper pump parts, pump calibration could not be completed until the latter half of December. Exploratory runs were performed the last week in December and continued through January and February of 1951.

Water was used to calibrate the pump. Inasmuch as the viscosity of cottonseed oil at operating temperatures, 2.74 centipoise at 330°F,[17] is not substantially different from water at room temperatures, it was felt that such a calibration would be well within experimental limits. Water was piped through a temporary 1-1/2 inch line from a constant head tank to the suction side of the pump, pumped through the system and into a drum on scales. The data were then converted to flow in gallons per minute. The calibration is presented in Figure 3.

Loading of the system was achieved using suction afforded by the recirculating gear pump. A rubber hose was connected to a tee on the suction side of the pump and block valves installed before the hose and on the recirculating line immediately following the connection to the product line from the filtrate tray. The hose was then placed in a drum of unhardened oil standing on scales and the gear pump operated until the proper weight had been taken from the drum, the

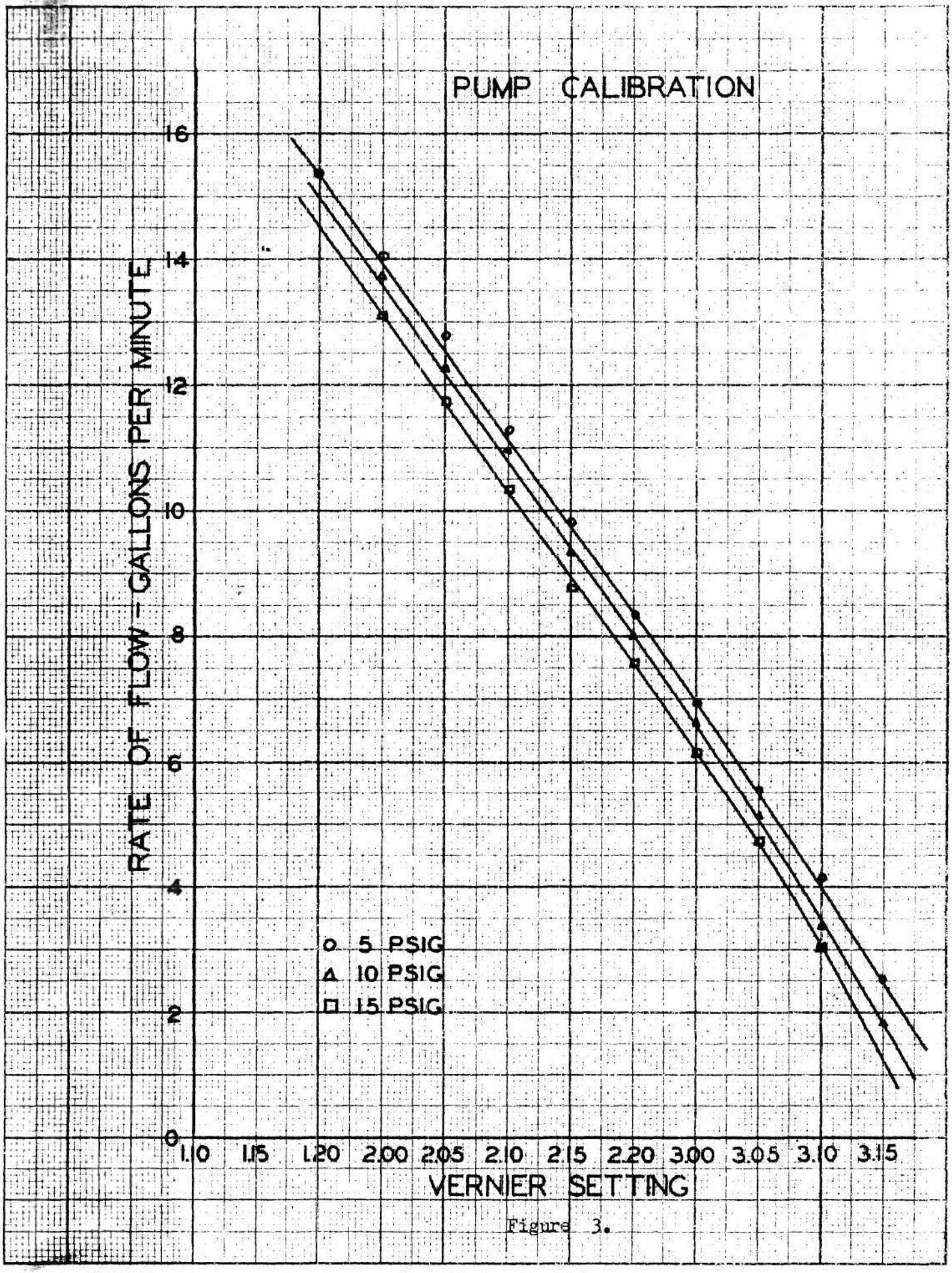
PUMP CALIBRATION
RATE OF FLOW - GALLONS PER MINUTE
16
14
12
10
8
6
4
2
0
o 5 PSIG
Δ 10 PSIG
□ 15 PSIG
1.10 1.15 1.20 2.00 2.05 2.10 2.15 2.20 3.00 3.05 3.10 3.15
VERNIER SETTING

Figure 3.

normal charge being 75 pounds. The main pump was also operated to prevent the surge tank from flooding. With the system pressure at atmospheric and the oil flowing at a rate of about four gallons per minute, the three electrical heaters were turned on. As can be seen with reference to the accompaning typical heating curve, the preheating required somewhat over four hours. During the charge-up and throughout the preheat nitrogen was kept bubbling through the system both to purge the system of air and to assure that the hydrogen jet should remain unplugged.

Before the end of the preheat period a portion of oil, about 700 cc, was removed and mixed with 200 grams of 28 per cent active metal catalyst and 100 grams of filteraid. This corresponds to about 0.16 per cent Ni in the total charge, a figure comparable to present day batch operation. While the catalyst slurry was being prepared the system was brought to operating conditions, adjusting the vernier by-pass and pressure regulating valve for the flow rate and pressure desired.

With the temperature up to 320°F, flow and pressure constant, the catalyst slurry was added to the surge tank through a funnel, access being gained through a tee in the recirculating line discharge to the top of the surge tank. After replugging the tee and allowing the oil to circulate for five minutes to assure a complete dispersion of the catalyst throughout the oil mass, the hydrogen was turned on and timing started.

Early attempts were made with a multiple-jet nozzle and a 2-5 lb/in^2 hydrogen pressure in excess over that of the system. However,

it was soon discovered that this led to agglomeration of relatively large bodies of gas in the oil stream. These gas bubbles, when expanded to atmospheric pressure through the pressure regulating valve, acted as a hammer on the surface of the oil in the surge tank, actually forcing oil through the open hydrogen vent line and out to the atmosphere. It was found that by using a single jet nozzle and a hydrogen pressure of 10-12 lb/in^2 in excess of system pressure a smooth operation of the process was obtained with the same rate of hydrogen flow.

Results

Data were taken at five minute intervals. Samples of oil for analysis were taken every ten minutes. Oil samples were taken from a sample cock located in the surge tank about an inch above the bottom. As it was necessary to filter samples rapidly and completely a special filtering routine had to be devised. After many trials, it was found most satisfactory to prepare a mixture of about equal portions of Dicalite and Santocel in a 500 ml Erlenmeyer, draw the sample from the surge tank into the flask, agitate well, and pour onto a Buchner funnel fitted into a suction flask connected to an air aspirator. After a portion of the oil had been drawn through and a cake formed, a test tube was placed under the neck of the funnel and the sample collected. The purpose of the Santocel was to adsorb colloidal or near colloidal catalyst particles which were apparently filtering through a cake of Dicalite alone. Filtration was also tried using Attaclay and activated charcoal but success was met only when Santocel was also used.

All heaters were left on until the system temperature reached 335°F. Operational data and samples were taken until completion of the run.

Upon completion of hydrogenation, the hydrogen was turned off and nitrogen again was injected into the system. All heaters were turned off and the oil continued to circulate at atmospheric pressure until the temperature dropped to about 250°F. The system was then set for filtration with only a partial closing of the block valve after the filter line outlet. The purpose of this procedure was to allow the press to warm up before filtration and prevent settling of catalyst in the system by maintaining flow.

By the time the press has warmed sufficiently, the oil temperature should be about 200°F, a safe temperature to begin filtration. If the oil does not begin to clear within a reasonable length of time more filter aid can be taken into the system by adding it in bulk at the press tray and drawing it with the oil through the recirculating line into the reservoir. As the oil supply in the system becomes depleted it becomes impossible to maintain pressure on the filter press. When the pump began to deliver air, as could be seen through the visible flow check valve, pressure was furnished by compressed air delivered through the blow lines. Valve arrangement permits blowing of the system either through the process piping, through the surge tank, or both. It must be emphasized here that steam is used for blowing only in case of fouled lines as water is a serious catalyst poison and the system must be thoroughly dried before using again.

Considerable difficulty was encountered in filtering every charge through the plate and frame press. This difficulty is attributed to colloidal catalyst particles present in the oil. Recirculation through the press up to five hours was carried on through cakes up to a half inch thick without clarification. Attempts were made to utilize Santocel as was done in collecting samples but the formation of a gel on the filter paper prevented use of sufficient quantity to effect separation.

Each sample was analyzed for iodine value according to the official methods of the American Oil Chemists Society.[18] All analytical reagents were prepared in strict accordance with these methods. It may be mentioned here, for the benefit of others who may do similar work, that the description of preparation of Wijs solution is somewhat inadequate. After preparation of the solution of iodine in glacial acetic acid, dry chlorine should be added with care. Chlorination should be stopped when a lightening of color of the solution first becomes apparent and not continued until a marked color change occurs.

The first few runs made were necessarily exploratory in nature in order to establish satisfactory process conditions. Although these first runs were invaluable as far as working out details were concerned, little applicable data were obtained.

Run E(see Table II) was performed with the system charged with 75 pounds of winterized cottonseed oil, 200 grams of 28 per cent active metal catalyst and 100 grams of Dicalite. System pressure was maintained at 10 lbs/in^2 and fluid flow was set at 5.15 gallons

per minute. The oil was preheated to 320°F and a hydrogen pressure of 20 lbs/in^2 was maintained into the jet. Hardening was attained reducing the iodine value from 107.5 in the original oil to 86.5 at the end of 80 minutes. This performance is comparable to present batch hydrogenation where similar reductions requiring two hours or more are common. An inspection of the iodine value versus time curve(see Figure 4) shows that only a very slight decrease in the rate of hydrogenation occurs as the reaction proceeds. This is particularly gratifying as it is often a point of industrial difficulty. Of a total of 38 cubic feet of hydrogen introduced into the system, 22.3 cubic feet actually reacted with the oil. This indicates a hydrogen efficiency of 58.7 per cent for a 47 foot length of such a process.

Run F(see Table III) was performed under identical conditions with the exception that a system pressure of 5 lbs/in^2 was used instead of 10 lbs/in^2. As would be expected, the rate of hydrogenation was considerably decreased, the iodine value dropping from 111.3 to 98.9 in 100 minutes. Of a total of 37 cubic feet of hydrogen introduced 13.2 cubic feet reacted with the oil, for a hydrogen efficiency of 35.6 per cent. As this loss of efficiency was more than should have justifiably occurred with such a pressure difference, it was suspected that the oil and catalyst were not agitated as much as the fluid flow settings indicated.

Run G(see Table IV) was performed under conditions identical with Run E. Although it was obvious from the beginning of the run that the pump was not delivering the fluid flow indicated, the run

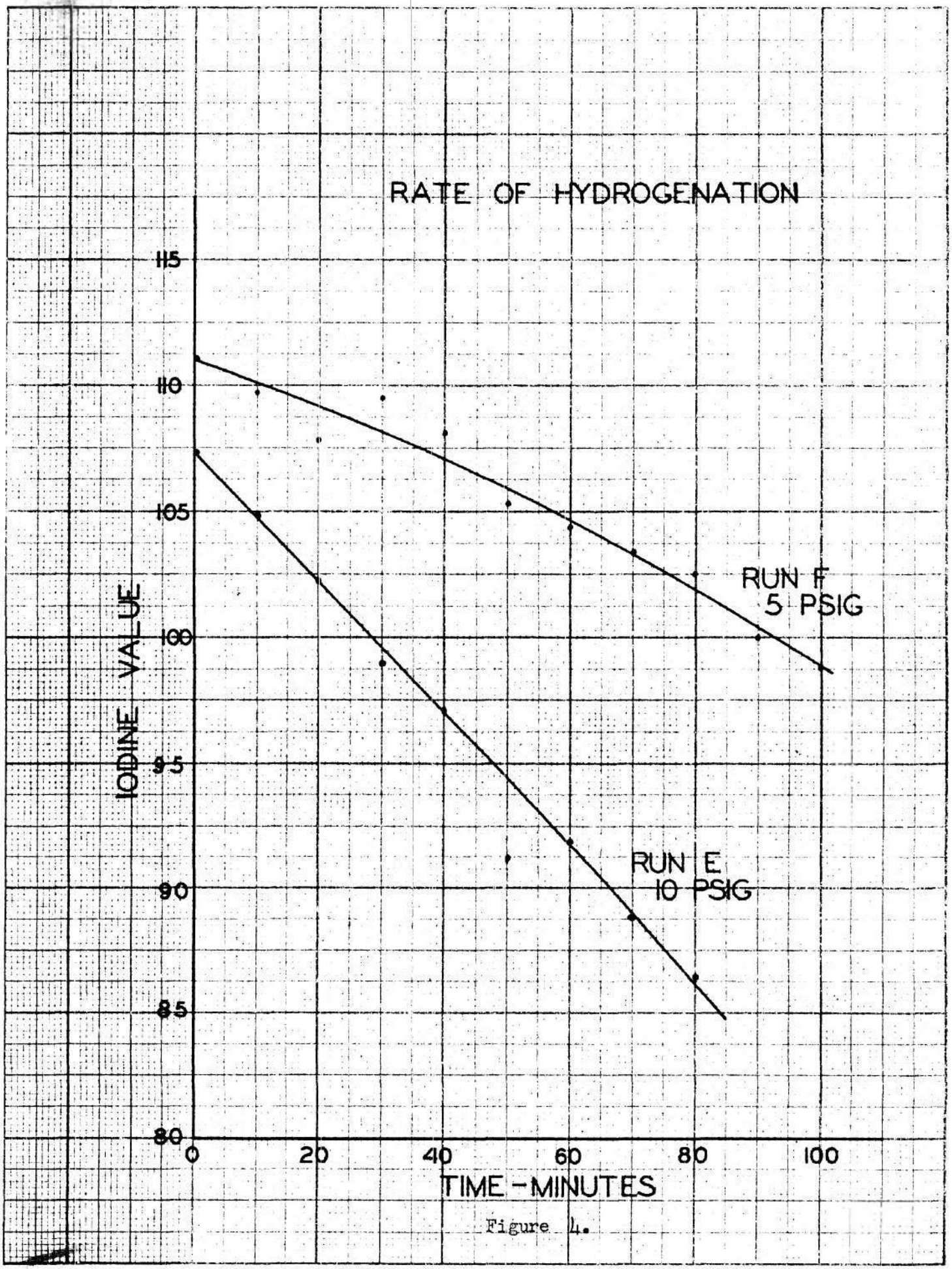
RATE OF HYDROGENATION
115
110
105
100
95
90
85
80
IODINE VALUE
RUN F
5 PSIG
RUN E
10 PSIG
0
20
40
60
80
100
TIME - MINUTES

Figure 4.

was continued. Subsequent tests at the completion of the run indicated that although the pressure remained fairly constant the actual flow of oil through the process piping was almost negligible. This condition afforded very poor contact between oil, catalyst, and hydrogen and poor results were expected. The iodine value was reduced from 113.7 to 105.0 in 110 minutes. Of a total of 33 cubic feet of hydrogen injected, 9.25 cubic feet were consumed by the oil giving a hydrogen efficiency of only 28 per cent.

Although the process as used represents a preliminary effort, it takes little imagination to see that in effect it is continuous, since it makes no difference whether it is recirculating around a short system or straight through a lengthy one. If we assume then, that all the hydrogenation that occurs, occurs between the point of injection of the hydrogen and the pressure reducing valve, we have a definite yard stick with which we can measure the length of process piping necessary to hydrogenate continuously to a given iodine value. This, of course, presupposes a hydrogen jet every 47 feet of process piping as well as comparable temperature, pressure, and fluid flow.

CONCLUSIONS

1. The work here reported indicates that a continuous process for hydrogenation of vegetable oils utilizing a two phase fluid flow in a pipeline is definitely feasible.

2. Further study should be made to establish optimum values of process variables, such as system pressure, fluid velocity in the pipe, rate of hydrogen injection, and preheat temperature.

3. Further work on the technique of sampling would definitely improve the quality of the data.

4. On the basis of Run E hydrogenation takes place at the rate of one iodine value for every 194 feet of reactor length. Undoubtedly this figure can be considerably reduced utilizing a larger reactor pipe and more favorable hydrogenation conditions.

5. It seems desirable to direct further work toward the development of a "differential reactor" in order that more reliable rate data and cost data may be obtained.

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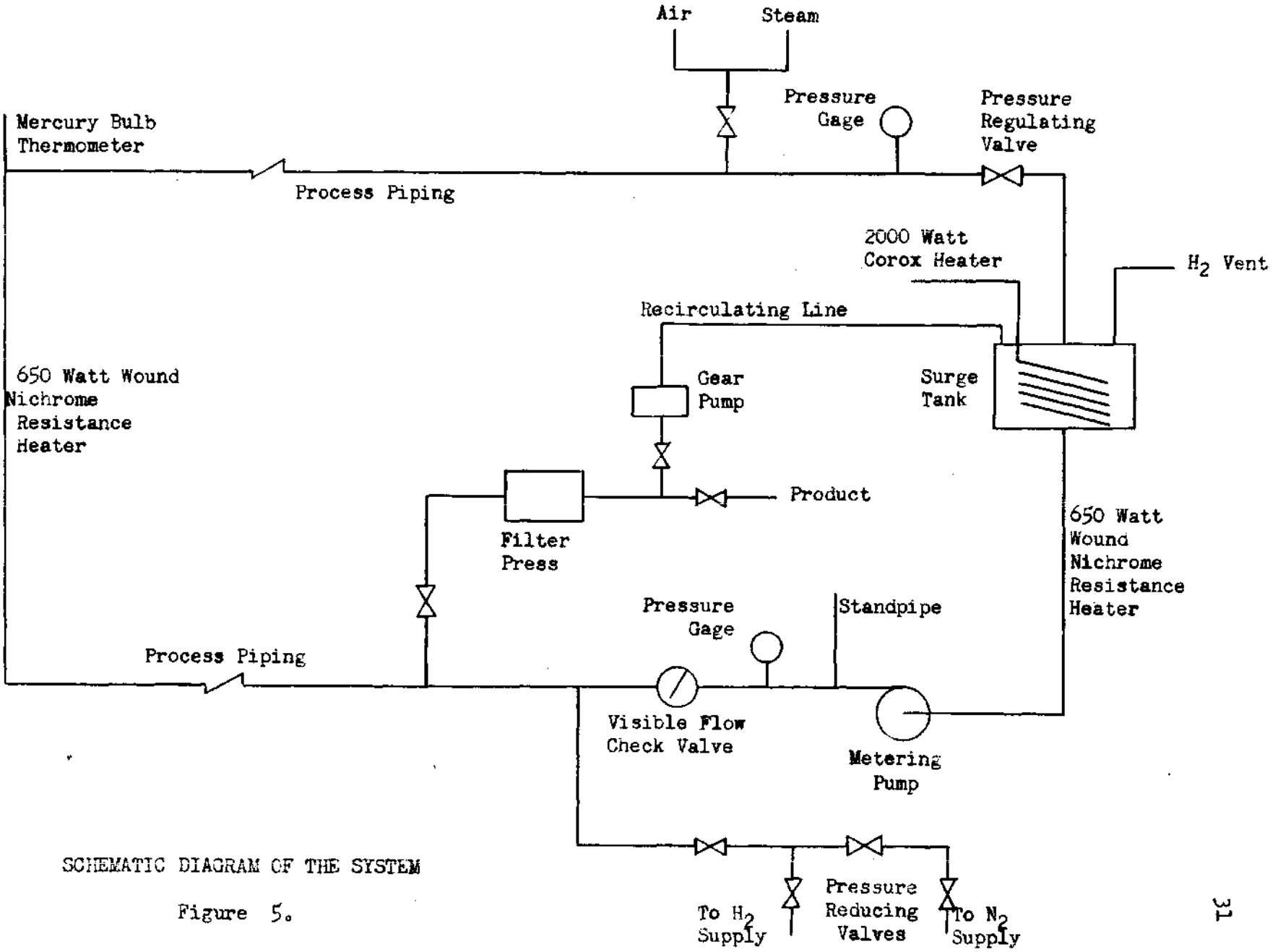
APPENDIX



SCHEMATIC DIAGRAM OF THE SYSTEM

Figure 5.

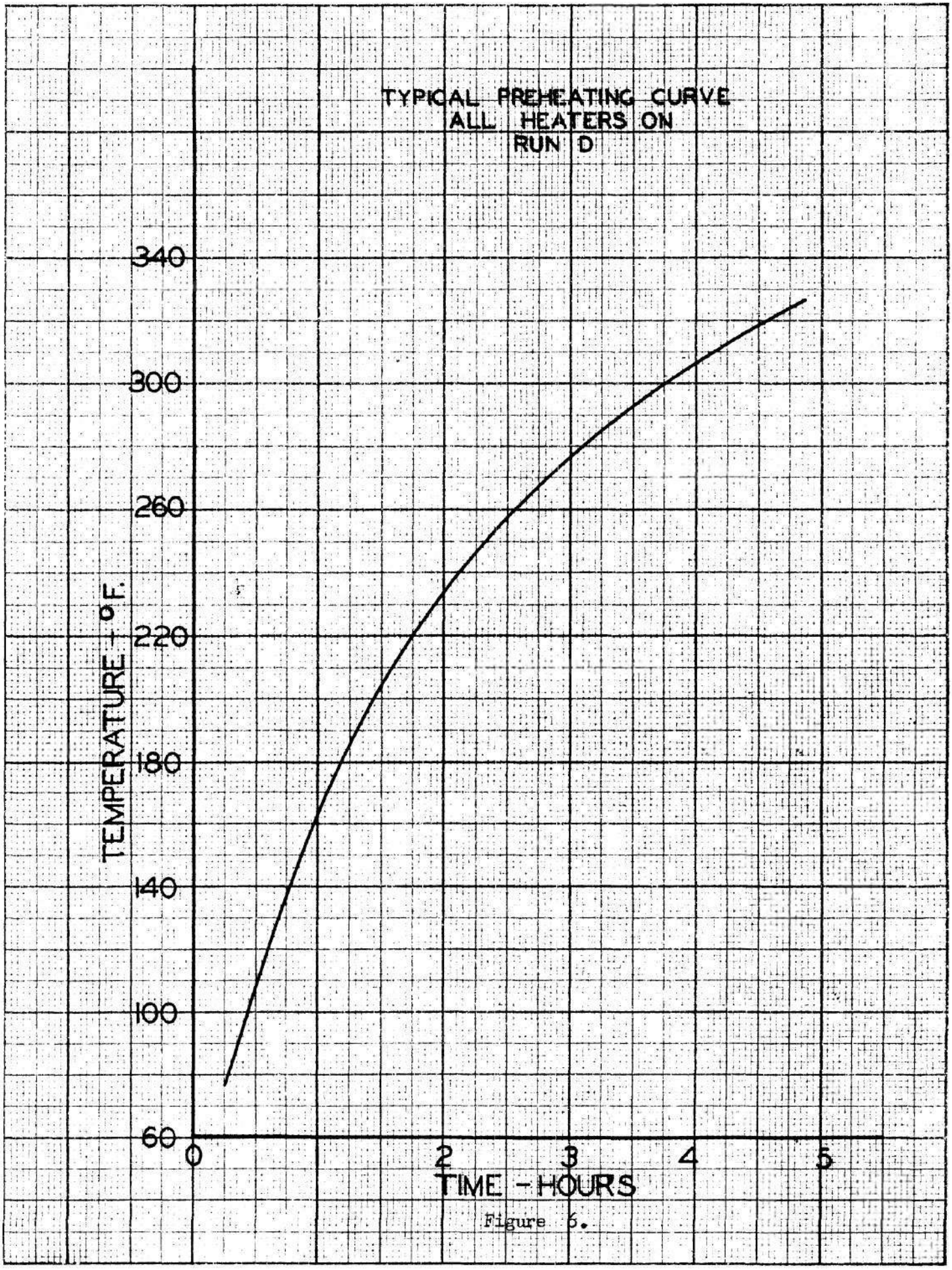

TYPICAL PREHEATING CURVE
ALL HEATERS ON
RUN D
TEMPERATURE - °F.
340
300
260
220
180
140
100
60
0
2
3
4
5
TIME - HOURS

Figure 6.

TABLE I

CALIBRATION DATA

Calibration	P_1 PSIG	Vernier Reading	Tare (Lbs)	Time (Min)	Weight (Lbs)	Weight (Lbs H_2O)	Wt. H_2O (Lbs/Min)	Rate (Gal/Min)
5 psig	8	2.00	7.6	0.75	95.5	87.9	117.2	14.03
	8	2.05	7.6	0.75	87.6	80.0	106.6	12.77
	8	2.10	7.6	1.00	102.0	94.4	94.4	11.30
	8	2.15	7.6	1.00	89.9	82.3	82.3	9.85
	7.5	2.20	7.6	1.50	112.1	104.5	69.7	8.35
	7.5	3.00	7.6	1.50	94.0	86.4	57.6	6.90
	7.0	3.05	7.6	2.00	100.0	92.4	46.2	5.53
	7.0	3.10	7.6	2.00	76.8	69.2	34.6	4.14
	7.0	3.15	7.6	2.00	50.0	42.4	21.2	2.54
	7.0	3.20	7.6	5.00	50.0	42.4	8.48	1.015
10 psig	12	3.00	7.4	2.00	118.5	111.1	55.55	6.65
	12	3.05	7.6	2.00	94.5*	86.9*	43.4*	5.19
	12	3.10	7.6	2.00	64.6	57.0	28.5	3.38
	12	3.15	7.6	2.00	39.0	31.4	15.7	1.88
	12	2.20	7.6	1.50	107.8	100.2	66.8	8.00
	12	2.15	7.6	1.00	85.7	78.1	78.1	9.35
	12	2.10	7.6	1.00	100.0	92.4	92.4	11.06
	12	2.05	7.6	1.00	110.0	102.4	102.4	12.28
	12.5	2.00	7.6	0.75	93.8	86.2	114.9	13.78
	13	1.20	7.6	0.75	104.3	96.7	128.9	15.41
15 psig	17	3.10	7.6	3.00	86.9	79.3	26.43	3.05
	17	3.05	7.6	3.00	126.6	119.0	39.66	4.75
	17	3.00	7.6	2.00	110.0	102.4	51.20	6.13
	17	2.20	7.3	1.50	101.8	94.5	62.9	7.54
	17	2.15	7.5	1.50	117.5	110.0	73.4	8.79
	17	2.10	7.6	1.00	93.7	86.1	86.1	10.32
	17	2.05	7.6	1.00	105.7	98.1	98.1	11.75
	17	2.00	7.6	0.75	89.7	82.1	109.4	13.11
20 psig	22	2.0	7.6	0.75	89.4	81.8	109.1	13.07
	22	2.10	7.6	1.00	92.9	85.3	85.3	10.22
	22	2.20	7.6	1.50	101.4	93.8	62.6	7.50
	22	3.00	7.6	2.00	108.5	100.9	50.5	6.05
	22	3.10	7.6	2.00	58.2	50.6	25.3	3.03
	22	3.05	7.6	3.00	116.7	109.1	38.4	4.59
*Alternative Values (check):					94.0	86.4	43.2	

TABLE II

RUN E OF 2/10/51*
75 Lbs. Winterized Cottonseed Oil (Swift)

Time (Min)	T_1 (°F)	P_1 (PSIG)	P_2 (PSIG)	PH_2 (PSIG)	H_2 (Ft^3)	Sample No.	IV Analysis
0	320	13	10	20	114	E0	107.5
5	320	14	11	20	111		
10	322	13	10	20	109	E1	104.9
15	324	14	11	20	107		
20	326	13	10	20	104	E2	102.3
25	327.5	13	10	20	102		
30	329	13	10	20	100	E3	99.0
35	331	13	10	20	98		
40	332	13	10	20	95	E4	97.0
45	334	14	11	20	92		
50	336	14	11	20	90	E5	91.1
55	337	13	10	20	88		
60	338	13	10	20	86	E6	91.4
65	339.5	13	10	20	84		
70	340.5	13	10	20	80	E7	88.8
75	342	13	10	20	78		
80	343	13	10	20	76	E8	86.5

*200 gms Selectol-A Cu-Ni catalyst (Drew).
100 gm Dicalite.
System pressure: 10 psig.
Vernier setting: 3.05 corresponding to fluid flow of 5.15 gpm.
Preheat temperature: 320°F.
Run made with single nozzle jet injector, 0.015" diameter.
Nozzle kept clear by continuous passage of nitrogen into system while preheating.

TABLE III

RUN F OF 2/17/51*
75 Lbs. Winterized Cottonseed Oil (Swift)

Time (Min)	T_1 (°F)	P_1 (PSIG)	P_2 (PSIG)	PH_2 (PSIG)	H_2 (Ft^3)	Sample No.	IV Analysis
0	322	8	5	15	73	F0	111.3
5	322.5	8	5	15	72		
10	326	8	5	15	70	F1	109.7
15	327	8	5	15	69		
20	330	8	5	16	66	F2	107.9
25	333	8	5	16	64		
30	332	8	5	16	62	F3	109.6
35	328	9	7	16	60		
40	325	9	7.5	16	58	F4	108.1
45	330	9	7	16	56		
50	331	9	7	16	54	F5	105.4
55	334	9	7	16	52		
60	336	9	7	16	50	F6	104.3
65	338	9	7	16	48		
70	340	9	6	16	47	F7	103.5
75	342	9	6	16	46		
80	341	9	6	16	44	F8	102.6
85	336	9	6	16	42		
90	334	9	6	16	40	F9	100.0
95	330	9	6	16	38		
100	329	9	6	16	36	F10	98.8

*200 gms Selectol-A Cu-Ni catalyst (Drew).
100 gms Attaclay.
System pressure: 5 psig.
Vernier setting: 3.05 corresponding to fluid flow of 5.15 gpm.
Preheat temperature: 322°F.
Run made with single nozzle jet injector, 0.015" diameter.
Nozzle kept clear by continuous passage of nitrogen into system while preheating.

TABLE IV

RUN G OF 2/25/51*
75 Lbs. Winterized Cottonseed Oil (Swift)

Time (Min)	T_1 (°F)	P_1 (PSIG)	P_2 (PSIG)	PH_2 (PSIG)	H_2 (Ft^3)	Sample No.	IV Analysis
0	319	10	9	15	226	G0	113.8
5	326	10	8	15	224		
10	328	10	9	15	221	G1	111.8
15	327	9	7	15	219		
20	333	11	9	15	218		
25	338	10	8	15	216	G2	110.6
30	337	10	8	15	214	G3	110.6
35	336	11	9	15	212		
40	334	10	9	15	210	G4	110.0
45	333	10	8	15	209		
50	333	12	10	17	208	G5	109.8
55	334	10	8	17	207		
60	333	9	7	17	205	G6	109.3
65	335	9	7	17	203		
70	335	12	10	17	201	G7	108.6
75	332	10	8	17	199		
80	333	10	8	17	197	G8	107.1
85	327	10	8	17	195		
90	322	9	7	17	193	G9	106.9
						G10	106.0

*200 gms Selectol-A Cu-Ni catalyst (Drew).
100 gms Attaclay.
System pressure: 10 psig.
Vernier setting: 3.05 corresponding to a fluid flow of 5.15 gpm.
Preheat temperature: 319°F.
Run made with single jet injector, 0.015" diameter.
Nozzle kept clean by continuous passage of nitrogen into the system while preheating.